

# Comparison of time-dependent changes in the surface hardness of different composite resins

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## ABSTRACT

**Objective:** The aim of this study was to evaluate the change in surface hardness of silorane-based composite resin (Filtek Silorane) in time and compare the results with the surface hardness of two methacrylate-based resins (Filtek Supreme and Majesty Posterior).

**Materials and Methods:** From each composite material, 18 wheel-shaped samples (5-mm diameter and 2-mm depth) were prepared. Top and bottom surface hardness of these samples was measured using a Vicker's hardness tester. The samples were then stored at 37°C and 100% humidity. After 24 h and 7, 30 and 90 days, the top and bottom surface hardness of the samples was measured. In each measurement, the rate between the hardness of the top and bottom surfaces were recorded as the hardness rate. Statistical analysis was performed by one-way analysis of variance, multiple comparisons by Tukey's test and binary comparisons by *t*-test with a significance level of  $P = 0.05$ . **Results:** The highest hardness values were obtained from each two surfaces of Majesty Posterior and the lowest from Filtek Silorane. Both the top and bottom surface hardness of the methacrylate based composite resins was high and there was a statistically significant difference between the top and bottom hardness values of only the silorane-based composite, Filtek Silorane ( $P < 0.05$ ). The lowest was obtained with Filtek Silorane. The hardness values of all test groups increased after 24 h ( $P < 0.05$ ). **Conclusion:** Although silorane-based composite resin Filtek Silorane showed adequate hardness ratio, the use of incremental technic during application is more important than methacrylate based composites.

**Key words:** Composite resin, silorane-based composite resin, surface hardness

## INTRODUCTION

The use of light-activated composite resins has increased considerably because they provide better esthetics and an opportunity to restore extensive restorations. The structure of resin matrices of polymerized composite resins comprises bisphenolglycidyl dimethacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEG-DMA) or urethane dimethacrylate (UDMA).<sup>[1]</sup> In the polymerization of resin-based composites, shrinkage occurs as a result of the change from carbon single to double bonds. This event, called polymerization shrinkage, is the largest cause of stress on the cavity walls, separating the composite material from the cavity walls. Separation of the composite material from the cavity walls reportedly causes microleakage

at the margins of the restoration and secondary caries, post-operative sensitivity and dental pulp pathology.<sup>[2-4]</sup> To overcome the problems associated with polymerization shrinkage, early attempts focused on the type and amount of the particles included in composite resins and on different applications to particle surfaces. Later studies focused on the relationship between the polymerization shrinkage and the monomers composing the organic matrix of composite resins.<sup>[5]</sup> For this reason, the 3M-ESPE Company developed the silorane matrix system, which differs from methacrylate-based monomers and released the first composite filler material in which this matrix system was used: Filtek Silorane. Silorane actually comprises two different monomers called siloxane and oxirane.<sup>[6]</sup> Siloxane increases the hydrophobic features of the composite and

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oxirane lowers the level of polymerization shrinkage compared with methacrylate-based composites. In addition, a polymerization reaction peculiar to this composite occurs on the monomer level.<sup>[7-9]</sup> The manufacturer has announced that this composite can be used easily on posterior Class I and II restorations. Polymerization of light-activated composite resins starts at the surface, where light is applied.<sup>[10,11]</sup> For this reason, sufficient polymerization cannot be provided toward the deepest parts of the restoration when using bulk technique. Insufficient polymerization may result in sensitivities, discoloration and the formation of marginal gaps in the restoration.<sup>[2]</sup> The degree of polymerization of resin-based restorative materials can be analyzed directly or indirectly using the different techniques. Direct methods such as laser Raman spectroscopy<sup>[12]</sup> and infrared spectroscopy<sup>[13]</sup> are complicated, expensive and time-consuming.<sup>[14]</sup> Indirect methods include techniques such as scraping,<sup>[15]</sup> visual evaluation,<sup>[16]</sup> and surface hardness.<sup>[17]</sup> Surface hardness is an accepted indicator of the polymerization degree and has been used in many studies. It is simpler than other techniques<sup>[18]</sup> and shows correlations with data gathered from the infrared spectroscopy method.<sup>[19-21]</sup>

Surface hardness of composite resins can be affected by factors such as the density and application time of the curing light as well as the structure, thickness and color of the material.<sup>[22-24]</sup> Ideally, surface hardness of the composite resin should be equal or close to equal throughout the restoration and for the life of the restoration. However, the surface hardness of composite resins differs over time.<sup>[25]</sup>

Many studies have analyzed the surface hardness of methacrylate-based composite resins with various techniques.<sup>[26-28]</sup>

The aim of this study was to evaluate the change in surface hardness of silorane- and composite resin-based restorative materials at different time intervals and compare the findings with the surface hardness of two different methacrylate-based composite resins.

## MATERIALS AND METHODS

The composite resins used in the study are shown in Table 1.

To evaluate the surface hardness of each composite material, 18 cylindrically shaped samples were prepared using 5 mm diameter and 2 mm deep Teflon molds. During the sample preparation, the Teflon molds were positioned over an acetate strip on a glass plaque. After composite resin insertion, a second acetate strip was placed on top of the mold with slight pressure to remove excess material from the mold. The composite materials were then light-cured with a light-emitting diode (LED) device (1000 mw/cm<sup>2</sup>) (Hilux, Benlioglu, Turkey) for 40 s according to the manufacturer's instructions and the acetate strips were removed.

The top and bottom hardness of the samples was measured using Vicker's hardness tester (HMV-II; Shimadzu, Japan). A 100-g load was applied through the indenter with a dwell time of 15 s. Measurements were performed 3 times for each sample at intervals of 0.5 mm. The first hardness measurements were then recorded. Following the first measurements, samples were stored at 37°C and 100% humidity for 24 h and 7, 30 and 90 days. Their surface hardness was re-measured and recorded. Afterward, by dividing the bottom surface hardness value by that of the top, the hardness ratio of the material was calculated.

**Table 1: The composite resins used in the study**

Material	Type	Shade	Content	Batch number	Filler content (%)	Filler particle size
Filtek Silorane 3M-ESPE, USA	Silorane based microhybrid composite resin	A2	Siloxane, oxirane, camphorquinone, iodonium salt, electron donor, quartz, yttrium fluoride, stabilizer and pigments	#8CN #9ET	55 (vol.)	0.1-2 µm
Filtek Supreme 3M-ESPE, USA	Methacrylate based nanofiller composite resin	A2	Bis-GMA, Bis-EMA, UDMA, TEGDMA, zirconia/silica	#5BW	59.5 (vol.)	Particle size = 20-75 nm, cluster size = 0.6-1.4 µm
Majesty Posterior Kuraray, Japan	Methacrylate based nanofiller posterior composite resin	A2	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, nano-sized alumina and glass-ceramic components, camphorquinone, accelerator and pigments	#00005B	82 (vol.)	Nanofiller: 20 nm, Microfiller: 1.5 mm

Bis-GMA: Bisphenolglycidyl dimethacrylate, Bis-EMA: Ethoxylated bisphenol-A dimethacrylate, UDMA: Urethane dimethacrylate, TEG-DMA: Triethylene glycol dimethacrylate

Statistical analysis was performed using the one-way analysis of variance, multiple comparisons were conducted using Tukey's test and binary comparisons were made by *t*-test at a significance level of  $P = 0.05$ .

## RESULTS

Mean top and bottom surface hardness values of the composite resins are given in Tables 2-4 and the change in surface hardness values over time is shown in Figure 1.

When the surface hardness values of the three composites were compared, only the silorane-based restorative material (Filtek Silorane) showed statistically significant differences ( $P < 0.05$ ).

The methacrylate-based composite Majesty Posterior showed the highest and the silorane-based composite showed the lowest microhardness values for both the top and bottom surfaces at all measuring periods.

The ratio of top and bottom surface microhardness values, which was termed the hardness ratio, varied between 0.85 and 0.98. The lowest hardness ratio was obtained from the silorane-based composite Filtek Silorane [Table 5]. Significant differences among the

hardness ratios of the three composite resins were detected ( $P < 0.05$ ).

When the correlation among Vicker's hardness values obtained immediately after polymerization and after 24 h and 7 and 30 days was analyzed, there was a statistically significant difference between the immediate and 24 h measurements on the top surface of the three composite resins. However, a significant difference between 24 h and 7 days and between 7 days and 30 days was observed only in the samples of the methacrylate-based composite Majesty Posterior ( $P < 0.05$ ). No significant differences were observed on the top surfaces of all three composite resins between 30 and 90 days ( $P > 0.05$ ).

When the difference based on time of the bottom surface hardness of the samples was analyzed, all immediate hardness values of the tested materials showed a significant increase after 24 h ( $P < 0.05$ ). The hardness values between 24 h and 7 and 30 days did not show a statistically significant difference ( $P > 0.05$ ).

## DISCUSSION

One of the most important factors affecting the clinical success of nanofiller composite resins, such as microfiller and hybrid composite resins is the materials polymerization throughout the restoration. Adequate polymerization is important in terms of the ideal physical and mechanical properties. As a result of inadequate polymerization, possible microlleakage at the margins of the restoration, discoloration, increased erosion, decreased mechanical strength, increased water absorption and decreased bonding strength have been reported.<sup>[29,30]</sup> In addition, the residual monomers released due to inadequate

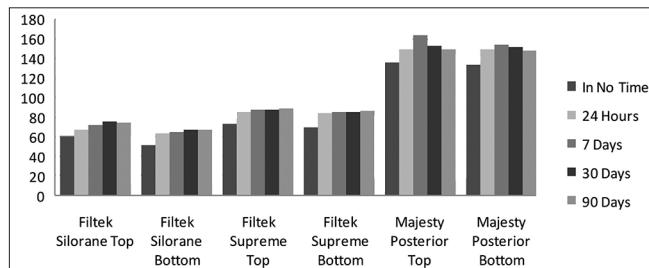


Figure 1: The change in surface hardness values over time

Table 2: Time-dependent change in the surface hardness of Filtek Silorane samples

Immediately		24 h		7 days		30 days		90 days	
Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom
58.72	50.16	66.6	62.77	71.01	64.63	74.32	66.52	73.78	66.9

Table 3: Time-dependent change in the surface hardness of Filtek Supreme samples

Immediately		24 h		7 days		30 days		90 days	
Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom
72.478	69.1	85	83.3	87.14	84.35	87.07	84.78	88.13	85.72

Table 4: Time-dependent change in the surface hardness of Majesty Posterior samples

Immediately		24 h		7 days		30 days		90 days	
Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom
135.5	132.83	149.05	148.722	163.39	153.611	152.22	150.5	149.16	147.33

**Table 5: The ratio of top and bottom surface microhardness values**

Material	In no time	24 h	7 days	30 days	90 days
Filtek Silorane	0.85	0.94	0.91	0.89	0.90
Filtek Supreme	0.95	0.98	0.96	0.97	0.97
Majesty Posterior	0.98	0.99	0.94	0.98	0.98

polymerization may pass through dentin tubules and cause irreversible damage.

Measurement of surface hardness is an indirect method of evaluating the polymerization degree of composite resin. Researchers have used different hardness measurement tests, such as those of Vicker, Barcoll and Knoop. Vicker's hardness test is easy to apply and the data obtained are reliable. The diamond indenter used in the procedure does not deform over time and is reportedly suitable for measurement of the hardness of fragile materials and dental tissue.<sup>[31]</sup> In the present study, the surface hardness of two methacrylate-based composite resins and one silorane-based composite was compared using Vicker's hardness measurement device.

The hardness of composite resin is reportedly affected by the color and depth of the composite material used as well as by the light device, period of light application and distance between the light tip and composite resin surface.<sup>[22-24]</sup> For this reason, the thickness of the samples was standardized to 2 mm in the present study. To avoid the effects of color on hardness, all three composites used in the study were of A2 color. All samples were polymerized using the same LED light device for 40 s, the light was implemented through a glass slab and the distance between the light device and the sample was anchored.

On both the top and bottom surfaces in all measurement periods, the highest hardness value was obtained in the samples of the methacrylate-based nanofiller composite Majesty Posterior and the lowest was obtained in the samples of the silorane-based composite Filtek Silorane. Guiraldo *et al.*<sup>[28]</sup> compared the top and bottom surface hardness of methacrylate- and silorane-based composite resins and found that the bottom surface value of the silorane-based composite resin was statistically significantly higher. Kusgöz *et al.*<sup>[32]</sup> have obtained similar results. These findings support the data obtained in this study.

There was a statistically significant difference among the hardness values of the composite resins used in this study. The reason for this difference is that the

monomer types, filler types and filler volume and polymerization mechanism of the composite resins used in the study differed.<sup>[29]</sup> In methacrylate-based composites, proximity of monomers react to establish a covalent bond in the polymerization process. On the other hand, the ring opening chemistry promotes expansion of the molecule during the polymerization process. The kinetics of the initiation and polymerization begin with cleavage and opening of the ring systems through a cationic ring opening reaction.<sup>[33]</sup> The resin matrix of one of the methacrylate-based composite resins, Majesty Posterior, comprises Bis-GMA and TEGDMA. Filtek Supreme, on the other hand, comprises Bis-GMA, TEGDMA, Bis-EMA and UDMA. The filler ratio of these materials is 82% for Majesty Posterior and 59.5% for Filtek Supreme volumetrically. The size of the filler particles is similar between the two.

In the silorane-based composite Filtek Silorane, which showed the lowest hardness degree, a silorane monomer was used. This type of monomer comprises siloxane and oxirane monomers in contrast to methacrylate-based composite resins. In addition, polymerization of this composite resin is quite different from the polymerization reaction of methacrylate-based composite resins. The main photoinitiator responsible for starting polymerization in methacrylate-based composites is camphorquinone. The reaction of this chemical with tertiary amines generates free radicals. In silorane-based composites, three main photoinitiators were used. These were camphorquinone, an iodonium salt and an electron donor. Camphorquinone was used because it is consistent with the emission spectra of the light used in the light devices. The electron donor, on the other hand, is come down to resolve the iodonium salt in an acidic cation and a split ring polymerization reaction is thus initiated.<sup>[7]</sup>

There were no significant differences between the top and bottom surfaces of the two methacrylate-based composites, whereas there was a significant difference between the top and bottom surface hardness of the silorane-based composite Filtek Silorane. Previous studies showed that the difference in hardness between the top and bottom surfaces of the composite resins was caused by inadequate light reaching the deep parts of the composite material.<sup>[29,32-35]</sup> In addition, while some of the light was absorbed by the composite material, some of it was shed.<sup>[32]</sup> This situation prevents the light from adequately penetrating the deeper parts of the composite material.

The top and bottom surface hardness values gathered from *in vitro* studies do not always indicate inadequate polymerization. For this reason, it is important to define a hardness ratio that states the ratio between the top and bottom hardness values in such studies. Ideally, these top and bottom surface hardness values are expected to be similar.<sup>[36]</sup> Pilo and Caardash<sup>[37]</sup> stated that the hardness value should be  $>0.80$ . All materials analyzed in this study had a hardness value of  $>0.85$ . This suggests that the LED light curing was adequate for polymerization of the 2 mm thick methacrylate and silorane-based composite samples.

In all three composites resins used in this study, the top and bottom surface hardness values differed significantly in the 1<sup>st</sup> 24 h. This is an indication that the polymerization reaction continues in the 1<sup>st</sup> 24 h. Previous studies have shown that microhardness values of composite resins are not constant and increase with time. Researchers suppose that unreacted free-radicals in the structure lead this event by continuing to generate cross-links after light application.<sup>[38-40]</sup> According to the day 7 data, the hardness values of all three composite resins decreased, followed by a relative increase. Our results support this information. Restorative material is affected by the saliva in the mouth and the food eaten. Ideally, the restorative material is expected to remain stable in the mouth. However, polymer-structured composite materials tend to absorb water and dissolve there in. Studies have shown a correlation between the water absorption and dissolving characteristics in water of composite resins.<sup>[41,42]</sup> Pereira *et al.*<sup>[43]</sup> analyzed the physicochemical features of restorative materials including different fillers and stated that when the filler amount increased, composite materials tended to absorb less water and exhibited lower solubility. The filler amount of the silorane-based composite resin Filtek Silorane was lower than that of the other two methacrylate-based composites. Yikilgan *et al.*<sup>[44]</sup> stated that the water absorption and water solubility of silorane-based composite resins were greater than those of the methacrylate-based composites. This might be why the silorane-based composite Filtek Silorane exhibited lower top and bottom surface hardness values and a lower hardening rate.

## CONCLUSION

Although silorane-based composite resin Filtek Silorane showed adequate hardness ratio, the use of incremental technic during application is more important than methacrylate based composites. Because

both the top and bottom surface hardness values of the silorane-based composite resin Filtek Silorane were lower than those of the methacrylate-based composite resins and there was a significant difference between the top and bottom surface hardness values of the silorane-based composite resin Filtek Silorane.

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